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## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In Re Application of

Maria Anna Wubben et al.

Serial No. 08/776,321

Examiner: C.E. Sherrer

Filed: April 15, 1997

Art Unit: 1761

FOT: PECTINS AS FOAM STABILIZERS FOR BEVERAGES HAVING A FOAM HEAD

## DECLARATION of Alexandra J.M. Wijsman

Alexandra Johanna Mathilda Wijsman herewith declares as follows:

- 1. I presently reside at Hondsdrafveld 23, 3448 EC Woerden, the Netherlands.
- 2. I am an employee of Heineken Technical Services B.V. at Zoeterwoude, the Netherlands, the assignee of the present patent application. I am a graduate of the Agricultural University of Wageningen, with a specialization in food technology. Since 1995 I have been involved in research on raw materials for the production of beer, such as polysaccharides.
- 3. Experiments were conducted in order to determine the effect of hop pectin and beet pectin on foam stability of beer. The experiments involved adding either hop pectin or beet pectin to a reference beer, which was a pilsner beer, and then measuring the foam stability in seconds.
- 4. The preparation containing hop pectin used in these experiments was obtained from CO<sub>2</sub> extract residus by an extraction procedure using the same



principles as described in the patent application on page 14, however, modified to take into account industrial size unit operations. The CO<sub>2</sub> extract residues were ground and mixed with warm acidified water. This suspension was filtrated to remove the solids. The extract obtained was mixed with alcohol to precipitate the pectin. The precipitate was separated from the solution and was washed out twice with alcohol after which the pectin was dried and an impure preparation containing hop pectin was obtained. In order to increase the purity of this hop pectin preparation, and hence to decrease the amount of non pectin components in this hop pectin preparation, it was subjected to an extra acidic alcohol washing step. Subsequently the preparation containing hop pectin was washed with alcohol to remove the acid. After drying, the hop pectin preparation was obtained.

- 5. The preparation containing beet pectin used in these experiments was a commercial beet pectin. This beet pectin is similar to the beet pectin used in the experiments described in the Declaration dated 13 July 1999, in said declaration designated as beet pectin 2. Commercial beet pectin is extracted from sugar beet pulp and is produced according to the classical hot, mild acid procedure. This hot, mild acid extraction procedure is also used for the extraction of hop pectins as described above. Since the beet pectin is a commercial product no further details of the production process are available.
- 5. To determine the effect of hop pectin and beet pectin on the foam stability of beer each pectin preparation was dissolved in 5 ml water before being added to a bottle of reference pilsner beer. The hop pectin preparation was 42.9% AUA, i.e. 11.7 g was added, thus providing 5g AUA per hectoliter of beer. The beet pectin preparation was 59.9% AUA, i.e. 8.4 g was added, thus providing 5 g AUA per hectoliter of beer. The bottles were gently shaken for 48 hours at room temperature. The foam stability was then determined using the Nibem foam meter.
- 7. The results of the foam stability experiments with hop pectin preparation and beet pectin preparation are presented in table 1.

	<del></del> _	Pectin addition as 5 g AUA / hi									
Samples	AUA content (%)	Foam number (sec)						Average foam improvement			
					111001	1	ауегада	(sec)	% relat d to reference		
Reference	<del>                                     </del>	265	266	263	267		265				
1010/0/100					254	000	350	85	32		
Hop pectin	42.9	361	331	345 311	351 310	362 306	303	38	14		
Beet pectin	59.9	295	295	1 211	1 210	1 500	1 000 1		•		

It is clear that the hoppectin provides a much better foam improvement than the beet pectin. These experiments were performed with the addition of hoppectin and the beetpectin at the end of the production process, i.e at a point of time where no substantial breakdown of the pectin takes place.

8. In the official action the Examiner considered that exp. 7 of table 2a of the previous declaration, is an aberration. Table 2a of this declaration is adapted by removing the data of exp.7. The result is as follows:

	F	oam number (se	Foam improvement (sec)		
Experiment	reference	hop pectin A	hop pectin B	hop pectin A	hop pectin E
1	257	288	349	34	89
	249	286	335		
2	252	290 332		37	82
	253	291	337		
3	237	288	307	50	82
	239	020			<u> </u>
4	276	321	363	37	86
	280	308	364		
5	275	323	353	47	79
	283	329	362		<b></b>
6	267	303	345	- 34	81
	282	314_	365	<del></del>	<del> </del>
average	263	302	345	40	83
stdev	16.7	16.0	17.6	6.9	3.7

It is clear that the removal of this one experiment does not change the conclusions that are drawn from this set of experiments.

9. The undersigned declares that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that wilful false statements so made are punishable by fine or imprisonment, or both, under Section 1001 of

Title 18 of the Unites States Code, and that such wilful false statements so made may jeopardize the validity of the document, or application, or any patent issuing thereon.

Signed this 22 day of November, 2001

Alexandra J.M. Wijsman